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IS 6890-1 (1989): Method for chemical analysis of dental gold alloys, Part 1: Determination of gold, silver, palladium and platinum [MTD 10: Precious Metals]



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“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

CHEMICAL ANALYSIS OF DENTAL GOLD ALLOYS

PART 1 DETERMINATION OF GOLD, SILVER, PALLADIUM AND PLATINUM

(*First Revision*)

भारतीय मानक

दांत के लिए स्वर्ण मिश्र धातुओं के रासायनिक विश्लेषण की पद्धति

भाग 1 स्वर्ण, चांदी, पॅलेडियम और प्लैटिनम ज्ञात करना

(पहला पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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FOREWORD

This Indian Standard (Part 1) (First Revision) was adopted by the Bureau of Indian Standards on 25 May 1989, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

Dental gold alloys in the form of wire, castings and dental white gold alloys, the compositions of which are covered in IS 3578 : 1985 'Specification for dental gold alloy wire (*first revision*)', IS 4799 : 1985 'Specification for dental casting gold alloys, non-porcelain bonding (*first revision*)', and IS 5954 : 1985 'Specification for dental white gold alloys (*first revision*)' are used in dentistry for various purposes. These alloys contain copper, nickel and zinc besides gold, silver and platinum group metals. The purpose of this standard is to lay down methods of chemical analysis which could be used for determining these elements in the ranges given in 1.1.

This part covers the methods of analysis for determination of gold, silver, palladium and platinum. The methods for nickel, zinc and copper are covered in Part 2.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF DENTAL GOLD ALLOYS

PART 1 DETERMINATION OF GOLD, SILVER, PALLADIUM AND PLATINUM

(First Revision)

1 SCOPE

1.1 This standard (Part 1) prescribes methods for chemical analysis of dental gold alloys having chemical composition within the following range:

Constituents	Percent
Gold	Above 28
Silver	3 to 30
Copper	2 „ 25
Palladium	0 „ 40
Platinum	0 „ 50
Zinc	0 „ 2
Nickel	0 „ 2

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	Title
IS 264 : 1976	Specification for nitric acid (second revision)
IS 265 : 1987	Specification for hydrochloric acid (third revision)

3 QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water shall be used.

4 GENERAL

4.1 Use of Filter Papers

In some of the methods prescribed in this standard, relative numbers of Whatman filter papers which are commonly used, have been specified. However, any other suitable brand of filter papers of corresponding porosity may also be used.

5 SAMPLING FOR CHEMICAL ANALYSIS

5.1 For Dental Alloy Castings

The pellets shall be hammered, rolled into a thin strip and cut into small pieces and a representative sample drawn.

5.2 For Dental Alloy Wire

The sample for chemical analysis shall consist of a composite sample of 10 g from each lot of 200 g or fraction thereof. Sample for this purpose shall be taken by shearing or clipping pieces from the entire cross-section, melting in a clean container at a temperature slightly above the melting range, mixing thoroughly and pouring into cold mould of convenient size. The sample so prepared shall be milled, drilled or sawed in such a manner as to represent the entire cross-section. Saw, drill or any other tool used shall be thoroughly clean and no lubricant shall be used in the operation.

6 DETERMINATION OF SILVER

6.1 Outline of the Method

The sample is dissolved first in nitric acid and then in *aqua regia* and silver determined as silver chloride.

6.2 Reagents

6.2.1 Dilute Nitric Acid, 1 : 1 and 1 : 99 (v/v).

6.2.2 Dilute Hydrochloric Acid, 1 : 4 (v/v).

6.2.3 Dilute Ammonium Hydroxide, 1 : 1 (v/v).

6.2.4 Aqua Regia

Mix one volume of concentrated nitric acid (sp gr 1.42) with three volumes of concentrated hydrochloric acid (sp gr 1.16).

6.3 Procedure (for Samples Containing Less Than 30 Percent of Gold)

6.3.1 Dissolve 1 g of an accurately weighed sample in 25 ml of dilute nitric acid (1 : 1) in a 250-ml beaker. Heat on a water-bath or hot plate and boil to expel brown fumes in a fuming cupboard. Cool, add 50 ml of water and allow to stand for one hour. Filter through Whatman filter paper No. 42, washing the precipitate several times with dilute nitric acid (1 : 99). Dry the filter paper, transfer to a crucible and reserve the residue for further treatment as given in 6.3.4.

6.3.2 Adjust the volume of the filtrate to 150 ml in a 250-ml conical flask and add from a burette, dilute hydrochloric acid drop by drop with constant stirring till the precipitation is complete. Add 1 ml in excess keeping it away from direct light. Heat, stir and allow to stand for about 2 hours in a dark place.

6.3.3 Filter through Whatman filter paper No. 42, wash the precipitate with warm dilute nitric acid (1 : 1) till washings are free from chloride. Reserve the filtrate and washings for treatment under 7.3.1.

6.3.4 Dissolve the silver chloride precipitate on the filter paper in dilute ammonium hydroxide solution and wash the paper thoroughly with hot water. Dry the filter paper and transfer to the crucible reserved under 6.3.1 and ignite.

6.3.5 Take the residue from 6.3.4 in a 250-ml beaker and add 25 ml of *aqua regia* and heat on a hot plate or a water-bath in a fuming cupboard. When the dissolution is complete, cool, and wash down the sides of the beaker, dilute to about 50 ml and allow to stand for 1 hour. If any precipitate of silver chloride appears filter through Whatman filter paper No. 42, and wash several times with dilute nitric acid (1 : 99). Reserve the filtrate and washings for treatment under 7.3.1. Dissolve the precipitate of silver chloride, if any, in ammonium hydroxide solution and wash the paper thoroughly with hot water.

6.3.6 Transfer the filtrate and washings obtained in 6.3.4 and 6.3.5 to a 400-ml beaker and acidify the filtrate with dilute nitric acid (1 : 1) with constant stirring, adding 1 ml in excess. Stir for some time and allow it to stand for overnight in a dark place.

6.3.7 Filter through a weighed sintered glass crucible of porosity No. 3. Wash the precipitate with warm dilute nitric acid (1 : 99) till free from chloride.

6.3.8 Dry the crucible and the precipitate at 120 to 130°C for about 1 hour. Cool in a desiccator and weigh.

6.4 Calculation

$$\text{Silver, percent} = \frac{A}{B} \times 75.26$$

where

A = mass in g of silver chloride, and

B = mass in g of the sample taken.

6.5 Procedure (for Samples Containing More Than 30 Percent of Gold)

6.5.1 Transfer 1 g of an accurately weighed sample to a 250-ml beaker and add 25 ml of dilute nitric acid (1 : 1). Heat on a water-bath or hot plate in a fuming cupboard till the reaction ceases. Decant off the solution and washings and keep the solution for further treatment.

6.5.2 Add 25 ml of *aqua regia* to the residue in the beaker and heat on a hot plate or water-bath, scraping off the coating of silver chloride which is formed on the sample, from time to time. Add more of *aqua regia*, if necessary. When the dissolution is complete, cool, wash down the sides of the beaker and add to the solution reserved under 6.5.1. Adjust the volume to 150 ml and add drop by drop dilute hydrochloric acid, if necessary to precipitate all the silver. Heat, stir well and allow to stand for about 2 hours in a dark place. Complete the determination as described under 6.3.3 to 6.4.

7 DETERMINATION OF GOLD

7.1 Outline of the Method

Gold in the presence of platinum and palladium is precipitated from the solution with sodium nitrite. The precipitate is filtered, washed, dried, ignited and weighed.

7.2 Reagents

7.2.1 Sodium Chloride, solid.

7.2.2 Sodium Nitrite Solution, 10 percent (m/v).

7.2.3 Dilute Sodium Nitrite Solution, 1 percent.

7.2.4 Concentrated Hydrochloric Acid, sp gr 1.16 (conforming to IS 265 : 1976).

7.2.5 Dilute Hydrochloric Acid, 1 : 5 (v/v).

7.2.6 Sodium Hydroxide Solution, 10 percent (m/v).

7.2.7 Thymol Blue Indicator

Triturate 0.1 g of the indicator in a clean glass mortar with 2.2 ml of sodium hydroxide (0.1 N) and then dilute to 100 ml with water.

7.2.8 Concentrated Nitric Acid, sp gr 1.42 (conforming to IS 264 : 1976).

7.3 Procedure

7.3.1 Take the solution reserved under 6.3.3 and 6.3.5 in a 400-ml beaker and add 0.1 g of sodium chloride. Evaporate to a syrupy consistency on a water-bath. Add 10 ml of concentrated hydrochloric acid and again evaporate to a syrupy consistency. Again add 1 to 2 ml of concentrated hydrochloric acid, 1 to 2 drops of thymol blue indicator, 100 ml of hot water, and stir.

7.3.2 Add sufficient sodium hydroxide solution to reach the red end point of thymol blue indicator ($pH=1.2$ to 2.8). Heat to boiling and add 10 ml of sodium nitrite solution. Boil the solution for 15 to 30 minutes to coagulate the gold maintaining the pH with small additions of sodium hydroxide solution. Add 20 ml of sodium nitrite solution and neutralize the hot solution with sodium hydroxide solution to reach the blue end point of thymol blue indicator ($pH=8$ approximately). Boil the solution for 5 minutes and add some more sodium hydroxide solution to restore the blue end point ($pH=8$ approximately). Allow the precipitate of gold, copper and zinc to settle by heating on a water-bath at 60°C for 15 minutes.

7.3.3 Filter the solution through Whatman filter paper No. 42 previously washed with dilute sodium nitrite solution and made alkaline to the blue end point of thymol blue indicator. Wipe the sides of the beaker with a piece of moist filter paper and transfer to the filter. Wash the precipitate with 50 ml of dilute sodium nitrite solution. Reserve the filtrate and washings for treatment under 8.3.1.

7.3.4 Wash the precipitate with hot water to ensure the complete removal of sodium nitrite. Dissolve the base metal precipitate in dilute hydrochloric acid and then wash with hot water. Place the filter in a porcelain crucible and dry in an oven.

7.3.5 Treat the filtrate again as under 7.3.2. Combine this second nitrite filtrate with the filtrate reserved under 7.3.3 for treatment under 8.3.1. Treat the reprecipitated base metals with dilute hydrochloric acid as given under 7.3.4 and add any small gold residue together with the paper to the first gold precipitate reserved under 7.3.4. Reserve the solution for treatment under 9.3.1.

7.3.6 Ignite the paper on a burner and then heat it in a muffle furnace at 800°C till complete incineration of the filter. Cool, add 5 ml of water and 2 to 3 drops of concentrated nitric acid. Heat for complete dissolution of sodium salts. Filter, wash with hot water, dry, ignite, cool and weigh (repeat ignition till constant mass is obtained).

7.4 Calculation

$$\text{Gold, percent} = \frac{A \times 100}{B}$$

where

A = mass in g of gold, and

B = mass in g of the sample taken.

8 DETERMINATION OF PLATINUM

8.1 Outline of the Method

Platinum is precipitated from the filtrate under 7.3.5 with ammonium chloride as ammonium chloroplatinate $[(\text{NH}_4)_2 \text{PtCl}_6]$ and ignited and weighed as metallic platinum.

8.2 Reagents

8.2.1 Concentrated Hydrochloric Acid, (see 7.2.4).

8.2.2 Hydrogen Peroxide, 30 volumes.

8.2.3 Ammonium Chloride, solid.

8.2.4 Dilute Ammonium Chloride Solution, 20 percent (m/v).

8.3 Procedure

8.3.1 Evaporate the filtrate reserved under 7.3.5 to dryness. Add concentrated hydrochloric acid in small portions to decompose the nitrite and again evaporate to dryness. Again add 5 ml of concentrated hydrochloric acid and evaporate. Repeat this treatment to remove oxides of nitrogen completely.

8.3.2 Add 2 ml of concentrated hydrochloric acid and 25 ml of water, and heat to boiling. Add 10 ml of hydrogen peroxide and boil for 15 minutes. Cool to 80°C, add ammonium chloride to form a saturated solution and stir. After stirring, allow the solution to stand in a cool place for overnight.

Filter the precipitate through filter paper No. 42 and wash with dilute ammonium chloride solution five times, keeping the precipitate covered with the wash solution throughout. Reserve the filtrate and washings for treatment under 9.3.1.

8.3.3 Fold the paper compactly about the precipitate and place in a porcelain crucible so that three-fold section of the paper is uppermost. Dry at 70°C for about 2 hours, increase the temperature slowly to 230°C and keep at this temperature for 3 hours. Then transfer the crucible to a muffle furnace maintained at 200°C, increase the temperature slowly and maintain it at 800°C for 1 hour. Cool in a desiccator and weigh as metallic platinum.

8.4 Calculation

$$\text{Platinum, percent} = \frac{A \times 100}{B}$$

where

A = mass in g of platinum, and

B = mass in g of the sample taken.

9 DETERMINATION OF PALLADIUM

9.1 Outline of the Method

In the filtrate from 8.3.2, palladium is precipitated with dimethyl glyoxime from the chloropalladium solution; filtered, dried and weighed as palladium dimethyl glyoxime $[(\text{CH}_3)_2 \text{C}_2\text{N}_2\text{O}_2\text{H}]_2\text{Pd}$.

9.2 Reagents

9.2.1 Concentrated Hydrochloric Acid, (see 7.2.4).

9.2.2 Sodium Salt of Dimethyl Glyoxime Solution

Dissolve 0.5 g of the reagent in 50 ml of boiling water.

9.3 Procedure

9.3.1 Take the solution reserved under 7.3.5 in 250-ml beaker, reduce the volume by evaporation to 5 ml to remove excess acid. Transfer the solution reserved under 8.3.2 to the same beaker and dilute to a suitable volume in volumetric flask. Take an aliquot of the solution containing 5 to 25 mg of palladium, add 5 ml of concentrated hydrochloric acid and dilute to 200 ml. Precipitate the palladium with 50 ml of filtered and hot dimethyl glyoxime solution. Stir well and allow it to settle in a warm place for about 30 minutes. Filter the solution containing palladium dimethyl glyoxime precipitate over sintered glass crucible No. 3 or Gooch crucible, wash with hot water and discard the filtrate.

9.3.2 Dry the crucible and precipitate at 110°C till constant mass, cool in a desiccator and weigh.

9.4 Calculation

$$\text{Palladium, percent} = \frac{A}{B} \times 31.68$$

where

A = mass in g of palladium dimethyl glyoximate calculated in the aliquot taken, and

B = mass in g of the sample taken.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg,
NEW DELHI 110002

Telephone

{ 331 01 31
331 13 75

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola,
CALCUTTA 700054

36 24 99

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036

{ 2 18 43
3 16 41

Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113

{ 41 24 42
41 25 19
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Western : Manakalaya, E9 MIDC, Marol, Andheri (East),
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